U. S. NAVAL AIR ENGINEERING CENTER

PHILADELPHIA, PENNSYLVANIA

AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC-AML-2503 DATE 7 September 1966

REACTIVATION OF CHROMATED CONVERSION COATINGS FOR MAXIMUM PAINT ADHESION

ASSIGNMENT 12-79(11D) UNDER NAVAIRSYSCOM AIRTASK
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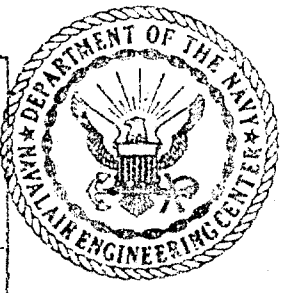

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ABSTRACT

Aircraft manufacturers have had considerable difficulty with adhesion of the epoxy paint system to aged chromated aluminum surfaces. This report presents results of an investigation of various methods to reactivate such surfaces and recommends several procedures that were effective in improving paint adhesion.

I. INTRODUCTION

A. Reference (a) requested an investigation to determine the cause of the poor paint adhesion being experienced by an aircraft manufacturer on new aircraft. Initially, the cause was believed due to contamination of the surface in the interim between chromating and painting. That the age of the chemical film at the time of painting might be a factor was also considered. The seriousness of the problem was verified by testing treated panels which were supplied by the manufacturer. The films were light in color and non-uniform in appearance; adhesion of paint to the film was very poor.

B. The investigation consisted of two phases:

1. tests designed to produce various types of contamination on chromated panels in an effort to duplicate the poor adhesion obtained with the manufacturer's panels;

2. procedures developed to clean or renew chromated surfaces and restore their efficacy as a paint base.

II. EXPERIMENTAL PROCEDURES AND RESULTS

A. Alloys and Finishing Systems

All testing in this study was carried out on 7075-T6 and 2024-T3 aluminum alloys, which were chromated with a MIL-C-5541A, Class 2 chemical film. Initial chromate treatments were dip applications (Grade C) in a 1.25 oz./gal. solution for approximately 3 minutes. Rechromating was accomplished with a brush application (Grade B) at a concentration of 4 oz./gal. These methods were applied in accordance with the vendor's recommendations. The paint system used was the MIL-P-23377 epoxy primer and the MIL-C-22750 epoxy topcoat without a wash primer. Prior to paint adhesion testing, the panels were room temperature aged for eight days. All paint adhesion testing adhered to paragraph 3.8.1 and 3.8.2 of MIL-C-5541A.

B. Phase I

1. Artificial Aging

It was theorized that the water content of chromate films was a possible cause for poor paint adhesion. A series of panels chromated at the Aeronautical Materials Laboratory (AML) were exposed to room temperature aging and artificial aging (120°F) for 48 hours, 96 hours, and 168 hours prior to painting, the aim being to determine whether dehydration of the chromate film could cause poor adhesion. Knife and wet tape tests showed that excellent adhesion was obtained on these panels, despite the fact that prior to painting the water content in the chemical film had been reduced.

2. Humidity Tests

In this test, efforts were made to increase the water content of chromated panels. Since most chromium compounds are hygroscopic, consideration was given to the possibility that the conversion coating would absorb moisture from the atmosphere on standing, and the paint adhesion be changed. Several panels treated at AML and an equal number treated by the manufacturer were subjected to 100% relative humidity at 120°F for 3 days. Equivalent sets were subjected to 50% relative humidity at 80°F. After painting, the panels treated by AML passed tape and knife tests on both the 50% and 100% relative humidity exposure panels, while the manufacturer's panels failed.

3. Contamination Tests

7075-T6 panels chromated at AML were stored in three separate locations for the period of one week in each location, starting with the Plating Laboratory, followed by the Salt Spray Room and the Machine Shop. These three locations provided acid and alkali fumes, salt-laden atmosphere, organic vapors, and dust and smoke from burning lubricants. After the exposure cycle was complete, these panels were painted without any solvent wiping, degreasing or cleaning of any kind. The tape and knife results proved to be excellent.

Six panels of the same alloy, chromate treated at the same time, were exposed in the welding area of the sheet metal shop for one month. After this time period, they were painted without any prior cleaning. The adhesion results again were excellent.

C. Phase II

Restoration Procedures

Since efforts to contaminate laboratory treated panels were unsuccessful, it was decided at this point in the investigation to abandon the search for the cause of poor paint adhesion on the manufacturer's panels. Efforts were then concentrated on finding cleaning methods that would restore good paint adhesion properties to aged and/or contaminated chemical films. In the next series of tests, both manufacturer's panels and panels chromated at AML were treated identically. Proprietary materials used are listed in the Appendix. The following procedures were investigated:

Procedure No. 1

Both groups of treated panels were cleaned with a MIL-C-5410B cleaner in the prescribed manner, rinsed and neutralized with the 5% by wt. aqueous solution of sodium bicarbonate. All of the manufacturer's panels thus treated showed water breaks after cleaning and after rechromating. Panels processed by AML gave the water break free surface. After painting, all panels passed knife and tape tests.

Procedure No. 2

A proprietary paste cleaner which had been recommended by another aircraft company for cleaning chromated surfaces was also investigated. Three formulations were submitted at varying times by the vendor.

a. Formulation A

Both groups were treated with this formulation diluted to 50% by wt. This material is brushed on to a thickness of between 8 and 10 mils. The treated panels were then allowed to dry as per vendor's instructions after which they were washed, rinsed, dried and painted. The manufacturer's treated panels did not have a water break free surface after cleaning, the AML panels did; however, tape and knife tests show good paint adhesion on both sets. Unfortunately, when this cleaner was used at the prescribed thickness on an aluminum assembly with cadmium plated steel fasteners, some of the cadmium plate was removed. If applied in excess of 10 mils, all of the cadmium plate is removed.

b. Formulation B

Two groups of panels were brushed with this material, allowed to dry for 2 hours, washed and rinsed. After paint adhesion tests, the manufacturer's panels gave poor results in the tape and knife tests. The panels treated at AML were satisfactory. Hydrogen embrittlement tests run on notched C-rings as per MIL-R-81294 showed this formulation was embrittling; one ring broke in 0.5 hours, another in 0.6 hours.

c. Formulation C

This material was tested under the same conditions as the other two above. The manufacturer's panels and those treated by AML showed good adhesion. This material, even applied in excess of 10 mils, did not remove cadmium plate from steel fasteners on an aluminum assembly and did not produce embrittlement in the notched C-ring test.

Procedure No. 3

In this test abrasive pads were used by hand with methyl ethyl ketone until all original chromate finish was removed. Both groups of panels were then rinsed and rechromated by brush application. All of the above panels gave a water break free surface. After rinsing, drying and painting, the knife and tape results proved to be excellent. This procedure did not remove a significant amount of cadmium plate.

Procedure No. 4

Another proprietary compound from another vendor was used as the cleaning medium in this test. Again both groups were brushed with this thixotropic material for 10 minutes, after which they were washed, rinsed, rechromated and painted. Adhesion tests show that each group had good knife and tape results, despite the fact that the manufacturer's panels did not have a water break free surface after cleaning or rechromating. On the aluminum assembly, this material removed all of the cadmium plate from the steel fasteners. Hydrogen embrittlement tests run with the notched C-ring specimen showed this material to be embrittling. One ring broke in 0.8 hours and the other in 1 hour.

Procedure No. 5

A water-emulsion cleaner had been recommended by the Overhaul and Repair Department, Alameda, California, as a solution to the problem. A material qualified under MIL-C-0022543C(WEPS) was diluted according to the manufacturer's instructions and scrubbed on the chromated aluminum surfaces of both groups of panels for 10 minutes. After rinsing, half of each group was chromated, rinsed, dried and painted, while the other half of each group was not rechromated but only rinsed, dried and painted. All panels originally chromated at AML gave excellent paint results whether they were rechromated or not. The manufacturer's panels that were not rechromated did very poorly; those that were rechromated gave excellent paint adhesion results.

Procedure No. 6

A large number of aluminum panels were chromated by immersion in the standard manner, rinsed, dried and then sprayed with a hand-strippable plastic coating (a one package system) to a thickness of .005 inch. These panels were then separated into several groups which were exposed for different lengths of time to natural sunlight; for 2 days, 2 weeks, 4 weeks, 8 weeks and 12 weeks. After exposure, these panels were hand stripped and painted without any precleaning or solvent wiping. Each group regardless of storage time or how long they were exposed to the natural sunlight gave excellent paint adhesion results.

III. DISCUSSIONS

The artificial aging experiments conducted clearly showed that the time interval between the application of a chemical film and the application of the paint system has no significant effect on paint adhesion if the film has been properly applied. The panels chromated at AML show no loss in adhesion whether the panels were room temperature aged or artificially aged. This experiment and its counterpart, the humidity tests, which were conducted to replace any moisture that might have evaporated from aged chromate films, are proof that moisture content has no significant effect on paint adhesion.

The storage tests conducted in the Plating Laboratory, Machine Shop, welding booth, and Salt Spray Room were designed to simulate aircraft surfaces exposed under shop conditions during manufacturing and assembly. When these panels were painted without any prior cleaning, the paint adhesion was excellent. This test was conducted only on panels chromated at AML under optimum laboratory conditions. It does prove, however, that a good chromate film is not particularly susceptible to contamination by vapors and fumes of the type investigated.

Cleaning Procedures

Before entering into a discussion of each cleaning procedure, it is interesting to note that Procedures 1 and 2 did not produce a water break free surface after the cleaning treatment on the manufacturer's panels and yet the paint adhesion was satisfactory. These results indicate that the presence of a water break does not necessarily mean the adhesion of the paint system will be poor.

In Procedure No. 1, cleaning with MIL-C-5410B cleaners produces satisfactory adhesion results as long as the requirements of the specification are met and the quality of the cleaning is maintained. In this procedure, the old chromate film was not removed by the cleaner. These panels were simply rechromated over the old film.

Paste cleaner Formulations A, B, and C used in Procedure 2, each have merit in a particular application. However, Formulation A, when applied in excess of 10 mils (which is not difficult to do when applied with a brush), removes cadmium plate from fasteners. Formulation B is an effective aluminum cleaner also, but this material is very embrittling to high strength steel. Formulation C is a good cleaner for aluminum and does not significantly remove cadmium plate or embrittle high strength steels. The use of this material results in excellent adhesion when applied in accordance with the manufacturer's instructions. Another advantage of this paste is that the original chromate film does not have to be rechromated after cleaning.

Procedure No. 3, abrasive pads used by hand with methyl ethyl ketone, was effective. Paint adhesion results from this method were excellent. Although there seems to be some question by other activities about the removal of cadmium plate from steel screws on aluminum assemblies and the inclusion of abrasive around the countersunk screws, the amount of cadmium plate removed is negligible compared to the overall advantage of providing excellent paint adhesion the first time an aircraft is painted. As far as ingredients are concerned, these pads are made up of aluminum oxide which is the abrasive, nylon mesh and a water insoluble resin which binds the two together. The uniform brushed surface produced by the use of these pads is also very beneficial because of the increased mechanical keying action which results with this surface, without the use of hazardous etching solutions of any kind. Opposed to all these advantages is one disadvantage, said to be considerable, and that is the cost of the labor involved in the hand operation.

In Procedure No. 4, another proprietary paste cleaner was used with reasonable success for removing the old chromate film and preparing the surface for rechromating. However, this material removes cadmium plate from the steel fasteners, and in addition, was found to be very embrittling to high strength steels. Because of these two deteriorative effects, this material cannot be used to reactivate old chromated surfaces on assembled aircraft structures.

In Procedure No. 5, the water emulsion cleaner gave excellent adhesion results on all the panels which were chromate treated at AML; but more important, it also produced excellent adhesion on the manufacturer's panels that were rechromated prior to painting. Although the aircraft manufacturer had also evaluated a cleaner conforming to the same specification, it had rejected its use because it did not produce a water break free surface. The product used at AML produced a water break free surface on the manufacturer's panels, but not on the AML panels. This material is not embrittling to high strength steels and does not effect the cadmium plate. This cleaner, because of the favorable paint adhesion results, and the ease of application, is another possible solution to the problem of obtaining good paint adhesion.

Procedure No. 6, utilizing the hand strippable plastic coating, seems to be another answer. Objections by an aircraft company that this type of film deteriorated on exposure to sunlight over a period of time appears groundless, at least up to three months. Adhesion of the epoxy system was not impaired by the use of this plastic coating, since after stripping, painting without any precleaning or solvent wiping, produced good results.

IV. CONCLUSIONS

A. If a chromate film is properly applied, the moisture content has no significant effect on paint adhesion; a good film is also not susceptible to contamination by vapors and fumes of the type investigated.

B. A water break free surface is not a prerequisite for good paint adhesion. Unfortunately, however, some other criterion to use as a substitute for determining a clean surface, has yet to be found.

C. The manufacturer's panels used in this investigation were produced under the quality acceptance requirements of MIL-C-5541, rather than MIL-C-5541A, since the more stringent requirements of the latter were not in effect until May 1966 when the first Qualified Products List was issued under the new specification. The conclusion is inescapable that the chemical film applied by the manufacturer was initially one of poor quality and if production samples had been tested under MIL-C-5541A, Class 2 requirements, it is unlikely that they would have passed. If current production can meet the new requirements, some of the problem may be eliminated.

D. Since several of the above procedures produced the required degree of paint adhesion, there is some choice as to which system to use. The procedures outlined above fall into two categories; one in which the original chromate film remains and the other where the film is cleaned or removed and the surface rechromated. If the original film was of poor quality, or has become badly scuffed, scratched and generally abused, the latter course is advisable. In either case, the following are procedures which can be used successfully.

1. Procedure No. 1 where the MIL-C-5410B cleaner and the neutralizing rinse are used in the prescribed manner with rechromating prior to painting;

2. Procedure No. 2 (c) in which Formulation C of a proprietary paste cleaner can be used with or without rechromating prior to the application of the paint system;

3. Procedure No. 3 which makes use of the abrasive pads. Though found objectionable by some activities, this method should be considered for use in problem areas where good paint adhesion is difficult to obtain;

4. Procedure No. 5 which makes use of a water-emulsion cleaner qualified under MIL-C-0022543C(WEPS);

5. Procedure No. 6 which uses a hand strippable plastic coating to protect the original chromate film so that rechromating is not required.

V. RECOMMENDATIONS

1. It is recommended that the aircraft manufacturers encountering this difficulty investigate the possibility of substituting one of the methods listed above for the manual method presently in use.

2. It is recommended that the manufacturers' initial chromating processes be carefully tested to determine whether they will meet the requirements of MIL-C-5541A, Class 2.

REFERENCES

(a) BUWEPS ltr RRMA-51:TAJ/46 of 22 Oct 1965

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13. ABSTRACT

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